USEPA REGION 9 LABORATORY
RICHMOND, CALIFORNIA

STANDARD OPERATING PROCEDURE 385

EXTRACTABLE PETROLEUM HYDROCARBONS BY GC/FID

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1 PURPOSE AND APPLICABILITY

This procedure describes the analysis dichloromethane extracts for total petroleum hydrocarbons (TPH) at the EPA Region 9 Laboratory. Gas chromatography (GC) with a flame ionization detector (FID) is used for the quantitative and qualitative determination of hydrocarbons. Water samples are prepared using EPA Region 9 Laboratory SOP 275 *Extraction of Water Samples by Continuous Liquid-Liquid Extraction*. Solid samples are prepared using EPA Region 9 Laboratory SOP 290 *Extraction of Soil Samples Using Pressurized Fluid Extraction.* Procedures for Soxhlet extraction or waste dilution to prepare other sample matrices for analysis are also available.

This procedure is applicable to the determination of TPH as diesel range organics, DRO:C_{10} - C_{24} and TPH as oil range organics, ORO:C_{24} – C_{40} in extracts prepared from solid or liquid samples. The procedure may also be used to determine other extractable petroleum hydrocarbon mixtures such as kerosene or hydraulic oil in these matrices. This SOP is based on procedures contained in EPA SW-846 method 8015C, Revision 3, February 2007. Deviations from the reference method are described in Appendix A.

Quantitation limits are provided in Appendix B by matrix and analyte.

2 SUMMARY

Sample extracts, which have been fortified with surrogate, are injected into a GC with FID. Sample components are separated in a fused-silica capillary GC column during temperature programming and detected by the FID.

Probable identification of fuels in samples is based on a comparison of the chromatographic pattern generated by analysis of the sample to the chromatographic pattern of known hydrocarbon fuel standards analyzed under the same conditions as the sample. The identification of specific fuel types may be complicated by substantial variation in fuel composition, and by environmental processes such as evaporation, biodegradation, or the presence of more than one fuel type. Data are qualified in a manner that reflects the qualitative uncertainty of the fuel identification.

The hydrocarbon concentration in the sample extract is determined by comparing the area sum response in the extract to the area sum response of hydrocarbon standards analyzed under the same conditions as the sample. The retention time range for the area response sum is determined from elution times of alkane hydrocarbons.

3 DEFINITIONS

FID - Flame Ionization Detector.

Laboratory Control Sample (LCS) - An aliquot of reagent water or other blank matrix to

which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine if the methodology is in control, and if the laboratory is capable of making accurate and precise measurements. The LCS is also known as a blank spike (BS).

LIMS - Laboratory Information Management System. The Element database.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD) - Two aliquots of the same environmental sample to which known quantities of the method analytes are added in the laboratory. The MS and MSD are treated exactly like a sample, and their purpose is to determine whether the sample matrix contributes bias to the analytical results and to indicate the precision associated with laboratory procedures. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS and MSD corrected for background concentrations.

Method Blank (MB) - An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, internal standards, and surrogates that are used with other samples. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

Method Detection Limit (MDL) - The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte.

Second Source Verification (SCV) - A solution of method analytes of known concentrations which are used to prepare mid level standard(s). The SCV solution is obtained from a source different from the source of calibration standards. It is used to check the accuracy of the initial calibration solutions.

Quantitation Limit (QL) - The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The QL is the concentration of the lowest non-zero standard in the calibration curve. Sample QLs are highly matrix-dependent.

Quantitation Limit Standard (QLS) - The lowest level CAL solution. The QLS is used to verify analytical system response at the quantitation limit.

Surrogate Analyte (SA) - A pure analyte which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in a known amount before extraction or other processing, and is measured with the same procedures used to measure other sample components. The purpose of the SA is to monitor method performance with each sample.

Stock Standard Solution (SSS) - A concentrated solution containing one or more method analytes purchased from a reputable commercial source.

TPH - Total Petroleum Hydrocarbons.

4 HEALTH & SAFETY

All laboratory operations must follow health and safety requirements outlined in current versions of the EPA Region 9 Laboratory Chemical Hygiene Plan and the Region 9 Laboratory Business Plan. Potential hazards specific to this SOP as well as pollution prevention and waste management requirements are described in the following sections.

4.1 Chemical Hazards

Due to the unknown and potentially hazardous characteristics of samples, all sample handling and preparation must be performed in a well-vented laboratory fume hood.

The toxicity and carcinogenicity of each reagent used in this method may not be fully established. Each chemical should be regarded as a potential health hazard and exposure to them should be minimized by good laboratory practices. Refer to the Material Safety Data Sheets located in Room 118 (library) and the LAN for additional information.

4.1.1 Dichloromethane

Dichloromethane is a suspected carcinogen. Effects of overexposure: acute inhalation or ingestion causes mild central nervous system depression. The primary toxic effect is narcosis. Other toxic effects are pulmonary edema, encephalopathy, and hemolysis. Dichloromethane irritates the eyes, skin, and respiratory tract. No systemic effects have been reported in humans, although excessive concentrations have caused cancer and liver and kidney damage in animals. Emergency and first aid - Inhalation: immediately remove to fresh air. If not breathing, administer mouth-to-mouth rescue breathing. If there is no pulse, administer cardiopulmonary resuscitation (CPR). Contact physician immediately. Eye contact: flush with water continuously for 15 minutes. Get emergency medical assistance. Skin contact: flush thoroughly for at least 15 minutes. Wash affected skin with soap and water. Remove contaminated clothes and shoes. Get emergency medical assistance. Ingestion: call local poison control center for assistance. Contact physician immediately. Never induce vomiting or give anything by mouth to a victim unconscious or having convulsions.

4.1.2 Acetone

Acetone liquid and vapors are highly flammable. Avoid heat, sparks, open flame, open containers, and poor ventilation. Effects of overexposure: Acetone is a mild eye and mucous membrane irritant, primary skin irritant, and central nervous system depressant. Acute exposure irritates the eyes and upper respiratory tract. Direct skin contact produces dermatitis, characterized by dryness and erythema through defatting of skin. High concentrations produce narcosis and hypoglycemia. Emergency first aid - Inhalation: immediately remove to fresh air. If not breathing, administer mouth-to-mouth rescue breathing. If there is no pulse, administer CPR. Contact physician immediately. Eye contact: flush with water continuously for 15 minutes. Get emergency medical assistance. Skin contact: flush thoroughly for at least 15 minutes. Wash affected skin with soap and water. Remove contaminated clothes and shoes. Wash clothing before re-use, and discard contaminated shoes. Get emergency medical assistance. Ingestion: call local poison control center for assistance. Contact physician immediately. Never induce vomiting or give anything by mouth to a victim unconscious or having convulsions.

4.2 Equipment and Instruments

Follow the manufacturer's safety instructions whenever performing maintenance or troubleshooting work on equipment or instruments. Unplug the power supply before working on internal instrument components. Use of personal protective equipment may be warranted if physical or chemical hazards are present.

Flame ionization detectors use hydrogen gas as fuel. If hydrogen flow is on and no column is connected to the detector inlet fitting, hydrogen gas can flow into the oven and create an explosion hazard. Detector fittings must either be capped or have a column connected at all times.

4.3 Pollution Prevention

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA Region 9 Laboratory places pollution prevention as the management option of first choice with regard to environmental management. Whenever feasible, laboratory personnel shall use pollution prevention techniques to address waste generation. When wastes cannot be feasibly reduced, recycling is the next best option. The *EPA Region 9 Laboratory Environmental Management System* provides details regarding efforts to minimize waste.

Minimize waste through the judicious selection of volumes for reagents and standards to prevent the generation of waste due to expiration of excess materials. Reduce the

volume of any reagent or standard described in Sections 7.2 or 7.3 so long as good laboratory practices are adhered to regarding the accuracy and precision of the glassware, syringes, and/or analytical balances used to prepare the solution. Reducing the concentration of a reagent is not allowed under this procedure because the impact of such a change on the chemistry of the procedure must be assessed prior to implementation.

Reduce the toxicity of waste by purchasing lower concentration stock standards, lower concentration stock reagents, and solutions to replace neat chemicals whenever possible. However, do not change the concentrations of standards and reagents specifically designated in this SOP

4.4 Waste Management

The EPA Region 9 Laboratory complies with all applicable rules and regulations in the management of laboratory waste. The laboratory minimizes and controls all releases from hoods and bench operations. All analysts must collect and manage laboratory waste in a manner consistent with EPA Region 9 Laboratory SOP 706 *Laboratory Waste Management Procedure* and City of Richmond Discharge Permit. Solid and hazardous wastes are disposed of in compliance with hazardous waste identification rules and land disposal restrictions. If additional guidance is needed for new waste streams or changes to existing waste streams, consult with EPA Laboratory Safety, Health, and Environmental Manager (LaSHEM) or ESAT Health and Safety and Environmental Compliance Task Manager or their designees.

This procedure produces the following waste streams:

Waste Stream Description	Waste Label	Hazard Properties
Laboratory solid waste (gloves, contaminated paper towels, disposable glassware, etc.)	Non-regulated Waste	Not applicable
Sample Extracts	Hazardous Waste	See solvent, diesel fuel and motor oil MSDs

5 SAMPLE HANDLING AND PRESERVATION

5.1 Internal Chain-of-Custody

- Sample extracts for GC analysis are received from the extraction lab personnel and custody transferred to the GC laboratory staff by signing the appropriate sections of the bench sheet. The extraction bench sheet and moisture determination records should accompany the sample extracts.

- The extracts are marked with the Region 9 Laboratory numbers and checked against the LIMS work order and chain-of-custody record to determine the client sample number, case number, and Sample Delivery Group (SDG) number.

5.2 Sample Extract Storage

- Store sample extracts in the refrigerator in Room 400 maintained at > 0°C to 6°C prior to analysis. Sample extracts must be analyzed within 40 days of extraction. Maintain a refrigerator temperature log daily. Report deviations following U.S. EPA Region 9 Laboratory SOP 805, *Refrigerator Temperature Monitoring*.

- Following analysis and reporting, the extracts must be stored under refrigeration for an additional 60 days before segregating for disposal. The sample results and preparation information are used to determine proper disposal.

6 INTERFERENCES

Chromatographic interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus that lead to anomalous peaks or elevated baselines in chromatograms, or by carryover when low concentration extracts are analyzed after high concentration extracts.

6.1 Extract contaminants

- Phthalate esters are commonly used as plasticizers and are easily extracted from plastic materials. Avoid contacting samples, solvents, reagents, glassware, extracts, or other sample processing apparatus with plastic materials.

6.2 Carryover

- Interfering contamination may occur when a sample containing low analyte concentrations is analyzed immediately after a sample containing relatively high analyte concentrations. Syringes and splitless injection port liners must be cleaned carefully or replaced as needed. After analysis of a sample containing high analyte concentrations, a laboratory instrument blank should be analyzed to ensure that accurate values are obtained for the next sample.

- Interfering contamination may occur when a sample containing oil range hydrocarbons, especially with carbon numbers exceeding C_{40}, is analyzed. After analysis of a sample containing oil range hydrocarbons, an instrument blank should be analyzed to ensure that accurate values are obtained for the next sample. The column may need to be heated to an elevated temperature, not exceeding the

column limit, until the baseline returns to previous levels. Syringes and splitless injection port liners must be cleaned carefully or replaced as needed.

7 APPARATUS AND MATERIALS

This section describes recommended apparatus and materials to be used for the analysis. All equipment, reagents, standards, and supplies must meet the technical and QC requirements of the reference method. Substitutions may be made provided that they are documented and equivalency is maintained.

7.1 Instrumentation

- Gas chromatograph with FID detector and splitless injection port (Agilent 6890, 7890, or equivalent).

- Fused silica capillary gas chromatography column -- Any capillary column with a phase ratio (β) of about 265 that provides adequate resolution and capacity may be used. The column used for method validation was 15M x 0.32 mm x 0.1 µm Rtx-1.

- Data acquisition and processing system -- software to control the GC and acquire, store, and process gas chromatographic data. The software must be able to calculate calibration factors and the concentrations of analytes in samples. Agilent Technologies EnviroQuant ChemStation software and data acquisition computers (or equivalent).

7.2 Reagents

- Acetone - capillary GC/GC-MS solvent grade.
  Caution: Acetone liquid and vapors are highly flammable. See Section 4.1.1 for precautions.

- Dichloromethane - recycled or capillary GC/GC-MS solvent grade.

  Caution: Dichloromethane is a suspected carcinogen. See Section 4.1.2 for precautions.

7.3 Standards

All standards must be entered into the EPA Region 9 laboratory information management system (LIMS).

- Surrogate Spiking Solution - Solution of n-hexacosane (n-$C_{26}H_{54}$) in dichloromethane:acetone 2:1 v/v at 2,500 µg/mL. Prepare from neat n-hexacosane

by weighing 125 mg n-hexacosane into a 50 mL volumetric flask, dissolving it in 33 mL of dichloromethane (may require sonication or warming) and diluting to volume with acetone.

- Instrument Blank - Solution of n-hexacosane in dichloromethane at 50 µg/mL. Prepare from the surrogate spiking solution by diluting 1 mL to 50 mL in dichloromethane.

- Stock Standard Solutions - Individual solutions of analytes purchased from commercial suppliers, such as Restek #31258 (Diesel Fuel #2 Composite Standard), or equivalent, or Restek #31256 (Kerosene Composite standard), or equivalent, or Restek #31464 (Motor Oil Composite Standard), or equivalent, or a homologous n-alkane series covering the carbon number range of interest. These solutions are diluted with dichloromethane to make the calibration solutions.

  Note: Whenever possible, the instrument should be calibrated using a sample of the fuel or oil that is contaminating the site. The calibration standard should be selected prior to the start of the project in conjunction with the client. A different calibration standard may be required if the fuel type in the sample does not match the calibration standard.

- TPH Matrix Spiking Solution - A solution of the fuel of interest at a concentration of 2,500 µg/mL in acetone. This solution is valid for six months from the date of preparation, or until ongoing QC indicates a problem exists, whichever is sooner.

- Calibration Verification Solution - Equivalent to the mid-point initial calibration solution.

- Quantitation Limit Standard (QLS) - Equivalent to the lowest level calibration standard. The QLS is used to verify instrument response at the quantitation limit.

- Second Source Verification (SCV) - Equivalent to the mid-point initial calibration solution but prepared from a source different from the source of calibration standards. The SCV is used to check the accuracy of the initial calibration solutions.

7.3.1 Calibration Solutions

Prepare TPH-DRO and TPH-ORO calibration solutions at five concentrations in dichloromethane from stock standard solutions at concentrations of 50,000 µg/mL and surrogate spiking solutions at concentrations of 2,500 µg/mL as shown in the tables below. All solutions are valid for six months from the date of preparation, or until ongoing QC indicates a problem. A standard containing homologous n-alkane series covering the expected range will also need to be

prepared at a concentration of 20 mg/L. Prepare by diluting 0.4 mL of Florida TRPH standard, Restek Cat. # 31266, to a final volume of 20 mL in dichloromethane.

TPH-DRO Solution	Volume Used, µL	Final Volume, mL	Final Concentration, µg/mL
Stock Standard	10	10	50
Surrogate Spike	40	10	10
Stock Standard	30	10	150
Surrogate Spike	100	10	25
Stock Standard	100	10	500
Surrogate Spike	200	10	50
Stock Standard	250	10	1,250
Surrogate Spike	300	10	75
Stock Standard	800	10	4,000
Surrogate Spike	400	10	100

TPH-ORO Solution	Volume Used, µL	Final Volume, µL	Final Concentration, µg/mL
Stock Standard	40	10	200
Surrogate Spike	200	10	50
Stock Standard	80	10	400
Surrogate Spike	200	10	50
Stock Standard	200	10	1,000
Surrogate Spike	200	10	50
Stock Standard	800	10	4,000
Surrogate Spike	200	10	50
Stock Standard	2000	10	10,000
Surrogate Spike	200	10	50

As an alternative to purchasing commercially available calibration solutions, standards may be prepared from neat fuels or oils as follows: Determine the density of the hydrocarbon fuel mixture by filling a tared 10 mL volumetric flask to volume with neat fuel at room temperature; record the weight in grams to the nearest 0.1mg. Divide the net weight by 10 mL to obtain the density in g/mL. Use the experimentally determined density in the following calculations.

Prepare a 4,000 mg/L (nominal) range standard by injecting 5 µL of neat standard per mL of dichloromethane. The actual concentration, in mg/L, will be 5,000 times the density of the neat standard in g/mL. For example, injecting 250 µL of kerosene into about 49 mL of solvent in a 50 mL volumetric flask, then adding additional solvent to volume, would result in a 3,910 mg/L standard assuming a density of 0.782 g/mL for kerosene.

If the neat standard, such as motor oil, is too viscous to measure with a microliter syringe, weigh out about 200 mg (0.2 g) using an analytical balance and dilute to 50 mL with dichloromethane.

Prepare the other calibration solutions by serially diluting the 4,000 mg/L standard.

7.3.2 Storage of Standard Solutions

Store the unopened ampulated stock standard solutions at > 0°C to 6°C. Store all other working standard solutions in glass bottles or vials with Teflon lined screw caps at ≤-10°C and protect all standards from light. Fresh standards should be prepared every six months, or sooner if comparison with check-standards indicates a problem. The standard solution must be checked frequently for stability. Replace all working standard solutions after six months or sooner if comparison with SCV indicates a problem.
CAUTION: Analysts must allow all standard solutions to equilibrate to room temperature before use. Hexacosane has poor solubility at low temperatures. Solutions containing hexacosane must be sonicated before use.

7.4 Supplies

- Volumetric flasks, type A, 100-mL, 50-mL, 25-mL, and 10-mL.
- Microliter syringes (10-µL, 25-µL, 50-µL, 100-µL, 250-µL, 500-µL, and 1-mL).

8 ANALYTICAL PROCEDURES

8.1 Instrument Operation

Set up the instrument operating parameters provided in Appendix D. Adjust as needed to meet method and SOP requirements.

Prior to analyzing calibration, QC, or field samples make a LIMS batch and sequence as required to obtain LIMS assigned IDs for the calibration and QC samples.

8.2 Calibration and Standardization

8.2.1 Initial Calibration

The initial calibration is a minimum 5-level external standard calibration using the mean CF for calculating sample analyte concentrations. See Section 9.2.1 of this SOP for required frequency and QC limits.

Prepare calibration solutions according to Section 7.3.1.

Analyze each of the initial calibration standards and an instrument blank using the same instrument conditions to be used analyzing field samples. Using the chromatography software, calculate the average calibration factors and %RSD using the following ranges: DRO:C_{10} - C_{24} and ORO:C_{24} – C_{40}. (See 8.3.3 for integration procedures).

8.2.2 Retention Time Windows

Based on experience and historical data, the EPA Region 9 Laboratory uses a default retention time window of +/- 0.03 minutes for single component analytes. This approach is based on the insignificant retention time drift observed historically and the option listed in SW846 Method 8000C section 11.6 to select an alternative approach to the usual analysis of standards for the calculation of retention time windows.

Establish the ChemStation retention time window as ± 0.06 minutes for the surrogate (peaks that drift more than 0.03 minutes will be flagged "f" by the data system as possible false positive).

8.2.3 Secondary Calibration Verification

- Analyze a SCV standard immediately after each initial calibration. See Section 9.2.3 of this SOP for frequency and QC limits.

  Note: Fuel standards from different sources may contain different compound mixes and therefore may not be reliable for verifying calibration standards.

8.2.4 Calibration Verification

Analyze a calibration verification standard in every12-hour analytical time period prior to an instrument blank analysis. The calibration verification standard is used to validate the initial calibration standard for the samples run during the associated 12-hour time period. The calibration verification standard concentrations are 500 µg/mL for TPH-DRO and 1,000 µg/mL for TPH-ORO. See Section 9.2.4 for calibration verification requirements and Appendix C for QC limits.

8.2.5 Quantitation Limit Standard

- Analyze a quantitation limit standard (QLS) each day when analyses of field or QC samples are performed. The QLS is used to verify analytical system response at the quantitation limit. The QLS is 50 µg/mL for TPH-DRO and

200 µg/mL for TPH-ORO. See Section 9.2.5 for QLS requirements and Appendix C for QC limits.

- If the initial calibration, the SCV, and the IB meet all the criteria specified in Appendix C, the remainder of the 12-hour analytical period may be used for the analysis of field and QC samples using the average CF from the initial calibration to quantitate the data.

8.3 Sample Analysis

8.3.1 Sample Preparation

Samples can be analyzed only after the initial calibration or calibration verification, QLS, MB, and IB meet all of the appropriate criteria specified in Appendix C.

Generate a LIMS batch and sequence as required prior to analyzing QC or field samples to obtain LIMS assigned IDs for the calibration and QC samples.

8.3.2 Analytical Sequence and Analysis

Set up a data acquisition sequence from the LIMS sequence using the GC operating parameters in Appendix D. The sample description shall include the laboratory sample ID. Additional header information should include the dilution factor, instrument ID, and the analyst's initials
Include all QC sample extracts. It is highly recommended that the MB, BS, and MS/MSD extracts be analyzed as early as possible in the analysis of a batch.

8.3.3 Analyte Identification and Quantitation

- After completion of analysis, review the chromatogram to identify the fuel in the sample. Compare the chromatographic pattern generated by analysis of the sample to the chromatographic pattern of fuels analyzed under the same conditions as the sample by visually comparing the printed chromatograms or by electronically overlaying the chromatograms, if needed. The diesel and oil ranges contain large number of chemical components which overlap. Use the following table in reporting the fuel and oil ranges:

Report	Chromatogram indicates the presence of:			
	TPH-DRO Only	TPH-ORO Only	Both DRO and ORO	Other components
DRO: C10-C24	Quantitate against the TPH-diesel range standard and report. Apply the appropriate LIMS flag to describe the fuel or product type (examples below).	Quantitate the overlap area against the TPH-diesel range standard and report the value as "non-detect" U.	Draw both integrations to the respective alkane markers and report both fuels Apply the appropriate LIMS flags for each to describe the fuel or product types (examples below).	Quantitate, the area in the DRO range. Apply the appropriate LIMS flag to describe the fuel or product type (examples below).
ORO: C24-C40	Quantitate the overlap area against the oil range standard and report the value as "non-detect" U. Apply the appropriate LIMS flag to describe the fuel or product type (examples below).	Quantitate against the oil range standard and report.	Draw both integrations to the respective alkane markers and report both fuels. Apply the appropriate LIMS flags for each to describe the fuel or product types (examples below).	Quantitate, area in the ORO range. Apply the appropriate LIMS flag to describe the fuel or product type (examples below).

LIMS Flag	**Description**
F0	[CUSTOM]
F1	Type: Not a fuel or hydrocarbon mixture.
F2	Fuel Type: Gasoline
F3	Fuel Type: Kerosene or Jet Fuel
F4	Fuel Type: Diesel
F5	Product Type: Motor Oil
F6	Product Type: Hydraulic Fluid
F7	Product Type: Lacquer Thinner
F8	Product Type: Mineral Spirits
F9	Product Type: Naptha
F10	Product Type: Stoddard Solvent
F11	Product Type: Turpentine

LIMS Flag	Description
F12	Single component, unidentified
F13	Fuel or Product Type: mixed or unknown

All detected results should be flagged in LIMS to qualify the reported value. When possible, indicate the product or fuel type. If a mixture is present, multiple flags may be appropriate (diesel and motor oil, kerosene and hydraulic fluid).

In the event that the client provides a fuel/product for calibration, the Technical Director will enter a project specific LIMS analysis. Use the F0 flag to indicate if the reported concentration resembles the standard used for calibration. If the sample contains a known product or fuel, use the appropriate flag, but quantitated against the client standard unless otherwise instructed. Describe the standard and calibration procedure in the WO memo field.

- Review the baseline drawn by the data system integrator to verify that it accurately reflects the area response of the fuel in the sample. If in the judgment of the analyst, it does not then draw a manual baseline from the elution time of C10 to the elution time of C24. See Appendix E for examples. Document any manual integration following the procedure described in U.S. EPA Region 9 Laboratory SOP 835, *Chromatographic Integration Procedures*.

- Quantitate the chromatogram using the appropriate initial calibration mean CFs for the identified fuel. If applicable, indicate degree of similarity of sample chromatogram to the fuel to which it is being compared. Print out quantitation reports and chromatograms for each field and QC sample.

- Water calculations

  Calculate results for target analytes using Equation 1:

  Equation 1:

$$\text{Conc. ug / L} = \frac{A_x \times V_t \times DF}{CF \times V_o}$$

  Where:

  A_x = area sum response of the sample
  DF = dilution factor
  CF = mean calibration factor from the initial calibration
  V_o = volume of water extracted in Liters
  V_t = volume of concentrated extract in mL

- Soil calculations

  Calculate results for target analytes using Equation 2:

  Equation 2:

$$\text{Conc. mg/kg (dry weight basis)} = \frac{A_x \times V_t \times DF}{CF \times W \times D}$$

  Where:

  A_x = area sum response of the sample
  D = dry weight factor (Percent solids/100)
  W = weight of sample in grams
  CF = mean calibration factor from the initial calibration
  V_t = volume of concentrated extract in mL
  DF = dilution factor

  Yields concentration units of µg/g = mg/kg

- Check surrogate recovery for each sample with criteria in Appendix C.

- Dilute and inject a new aliquot of the extract if the on-column concentration of the fuel of interest in any sample exceeds the initial calibration range. Use the following criteria in performing dilutions:

  1. Use the results of the original analysis to determine the approximate dilution factor required to get the fuel of interest within the initial calibration range.
  2. Do not dilute MS/MSD samples to get either the spiked or non- spiked target compounds within the initial calibration range. If the sample from which the spike aliquots were taken contains high levels of the spiked analytes, calculate the concentration and recovery of the analytes from the undiluted analysis, and note the problem in the report narrative.
  3. In the case of extremely contaminated samples several dilutions may be required.
  4. Distinguish between the undiluted and diluted analysis by adding a "REx" suffix to the client sample ID on the diluted analysis.
  5. Demonstrate that there is no carryover to subsequent analyses after a sample is analyzed that contains compounds at a level exceeding the initial calibration range of the system. This can be done by analyzing an instrument blank.
     Review the results for the sample analyzed immediately after a

contaminated sample for all compounds that were in the contaminated sample that exceeded the limits above. The sample should not contain a concentration above the QL for the target compound that exceeded the limits in the contaminated sample.

6. The most common cause of carryover is hydrocarbon in the oil/asphalt range. This may require cleaning the injection port and baking out the column.

8.3.4 QC Review

- Review results of instrument QC (CV, QLS) immediately after their analysis to verify that the results are within QC limits. If the instrument QC results are not within QC limits, stop the sequence and take corrective action before resuming the sequence. See Section 9.2; see Appendix C for QC limits.

- Review results of batch QC (MB, BS, MS/MSD). See Section 9.3; see Appendix C for QC limits.

- Review results of sample QC (surrogate recovery). See Section 9.4; see Appendix C for QC limits.

8.3.5 Data Export and LIMS Entry

Export data from the instrument into text files. Import into the LIMS using DataTool. Review final results in the LIMS.

LIMS will report all results to two significant figures and detected results to one-half the QL. LIMS flags values between one-half the QL and the QL as estimated (J); the analyst must manual add a "C1" flag to these results.

8.3.6 Instrument Maintenance

The following are suggested remedial actions that may improve method performance; re-calibration may be necessary after most of these actions:

- Check and adjust GC operating conditions and temperature programming parameters.

- Clean or replace the splitless injector liner with a new, silanized liner.

- Cut off a short portion of the GC column from the end near the injector, or replace the column. Cutting off a portion of the column will somewhat shorten the analyte retention times.

- Prepare fresh calibration solutions and repeat the initial calibration.

- Replace any components in the GC that permit analytes to come in contact with hot metal surfaces.

The analyst should observe trends in the data such as declining response, erratic relative response, loss of classes of compounds, etc., which may signal the need for instrument maintenance. Document all routine maintenance or corrective actions taken in the maintenance logbook. Preventative maintenance procedures are listed in Appendix F.

The following sections describe possible causes and corrective actions for common problems. Refer to Appendix F for routine preventative maintenance procedures and schedule.

Symptom

- Carryover
  Possible causes: Analyzing a sample containing high mole weight components or analyzing high-level and low-level samples sequentially.
  Corrective action: As necessary, replace inlet liner, clean inlet, bake out inlet, bake out column, clip column, replace septum, replace column.

- Shorter retention time.
  Possible cause: column flow rate problem.
  Corrective action: check flow rate and adjust as necessary.

- Longer retention time and or smaller peaks.
  Possible causes: column flow rate problem, injection port leak, or column contamination.
  Corrective action: As necessary, check for leaks, replace septum, replace the liner, replace the lower injection port seal, and cut the column (a few inches to a foot or more) from the injector end. If issues remain, replace the column.

- Loss of resolution.
  Possible causes: column flow rate problem, injection port leak, or column contamination.
  Corrective action: Check for leaks, replace septum, replace the liner, replace inlet seal, clip the column (a few inches to a foot or more) from the injector end. If issues remain, replace the column.

9 QUALITY CONTROL

9.1 Demonstration of Capability

The EPA Region 9 Laboratory operates a formal quality control program. As it relates to this SOP, the QC program consists of a demonstration of capability, and the periodic analysis of MB, LCS, and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data that are generated. A summary of the QC Criteria is provided in Appendix C.

A Demonstration of Capability must be in place prior to using an analytical procedure and repeated if there is a change in instrument type, personnel, or method. Follow procedures described in U.S. EPA Region 9 Laboratory SOP 880 *Demonstration of Laboratory Capability and Analyst Proficiency* for more details.

9.2 Instrument QC

9.2.1 Initial Calibration

Demonstration and documentation of an acceptable initial calibration are required before any samples are analyzed. The calibration is a five level external standard calibration method.

The GC system must be calibrated whenever corrective action changes instrument response (e.g., detector gas adjustment, column replacement, etc.) is performed or if the calibration verification criteria cannot be met.

- Analyze the alkane standard to determine the elution time for C10, C24, and C40.
- Analyze the initial calibration standards according to Section 8.2.1.
- Enter the carbon marker retention times into the method and obtain area sums for each fuel mixture in the designated time ranges.
- Draw a manual baseline if the baseline drawn by the data system integrator does not accurately reflect the total area response, including the unresolved area that lies below the individual peaks, of the fuel. For DRO draw a manual baseline beginning at the elution time of C10 to the elution time of C24. For ORO draw a baseline beginning at the elution time of C24 and going to the elution time of C40. Manual integrations must conform to U.S. EPA Region 9 Laboratory SOP 835, *Chromatographic Integration Procedures.* See Appendix E for example chromatograms.

- The data system calculates the calibration factor (CF) for the target fuel or n-alkane mixture from its area sum response and for the surrogate for all five calibration standards using Equation 3.

  Equation 3

  $$CF = (A_x)/ (C_x)$$

  Where
  Ax = Area of compound x
  Cx = Concentration of the standard injected (µg/mL)

- Calculate the average CF for all analytes.

- Calculate the percent relative standard deviation (%RSD) of the CF values for each compound using Equation 4.

  Equation 4

  $$\%RSD = (SD/ CF_{avg}) \times 100$$

  Where SD is calculated as:

  $$SD = \sqrt{\frac{\sum_{i=1}^{n} (CF_i - CF_{avg})^2}{n-1}}$$

- Verify that the %RSD of both the target fuel(s) and surrogate are within QC limits immediately after the initial calibration is finished. See Appendix C for QC limits.

- If an ICAL fails because of one standard, a fresh solution of that standard may be re-analyzed and substituted for the failed one in the ICAL. If more than one standard fails, corrective action is required.

9.2.2 Retention time windows

Retention time windows must be established when a new GC column is installed or when a new DOC is required.

- All surrogates in the field and QC samples must fall within the established retention time windows.

- All target analytes, surrogates, and/or n-alkanes in the calibration verification analyses need to fall within previously established retention time windows. If the retention time of any analyte does not fall within the established window, then corrective action must be taken to restore the system or a new calibration curve must be prepared for that compound.

9.2.3 SCV Analysis

Analyze an SCV sample immediately after each initial calibration. See Appendix C for QC limits. If the SCV sample fails it may be repeated once. If the second SCV fails, the cause for failure must be determined and corrected before analysis of samples can proceed.

Note: Fuel standards from different sources may contain different compound mixes and therefore may not be reliable for verifying calibration standards.

9.2.4 Calibration Verification

- Analyze a calibration verification standard at the beginning of each 12-hour analytical period and at the end of the 12-hour analytical period. The 12-hour analytical period begins with the injection of the calibration verification standard and ends with the completion of analysis of the last sample that can be injected within 12 hours of the beginning of the period. Analysis of calibration verification standards after every ten samples is recommended to minimize the amount of re-runs necessary if a calibration verification should fail. The calibration verification standard is used to validate the initial calibration for the samples run during the associated 12-hour time period.

- Analyze the calibration verification standard according to Section 8.2.4.

- Calculate the calibration factor (CF) for the target fuel from its area sum response and for the surrogate compound using Equation 3.

- Calculate the percent difference (%D) between the calibration verification CF and the initial calibration average CF for the target fuel and the surrogate using Equation 5.

Equation 5.

$$\%D = \frac{\mathrm{CF_c} \times \mathrm{CF_{avg}}}{\mathrm{CF_{avg}}} \times 100$$

Where:
$\mathrm{CF_c}$ = calibration verification CF

CF_{avg}= initial calibration average CF

- The %D must be within QC limits. See Appendix C for QC. If an analyte fails this criterion a second calibration verification may be analyzed. Repeated failure requires that corrective action be taken to restore the system before any additional samples are analyzed. All affected samples must be re-analyzed.

  If significant repairs to the system are required then a new initial calibration must be performed. The analyst should observe trends in the data such as declining response, erratic response, etc., which may signal the need for instrument maintenance.

- Acceptable sample analyses must be bracketed by the analyses of calibration verification standards that meet QC limits.

9.2.5 Quantitation Limit Standard (QLS)

- Analyze a quantitation limit standard (QLS) each day when analyses of field or QC samples are performed. The QLS is used to verify analytical system response at the quantitation limit. The QLS is analyzed at 50 µg/mL of TPH-DRO and 200 µg/mL for TPH-ORO.

- Analyze a standard of the fuel of interest at the concentration of the lowest initial calibration level according to Section 8.2.1 of this SOP.

- Calculate the concentration of the target fuel.

- Calculate the percent of true value for the target fuel using Equation 6.

  Equation 6:

  $$\% \text{ True Value} = (Cd/Tv) \times 100$$

  Where:
  Cd = Concentration determined by analysis
  Tv = True value of standard

- If the %D is not within the QC limits in Appendix C, a second QLS sample may be analyzed. Repeated failure requires that the cause be determined and corrected before analysis of samples can begin. If significant repairs to the system are required then a new initial calibration must be performed.

9.2.6 Instrument Blank (IB)

- Analyze an instrument blank at the beginning of each analytical sequence, or after any high level sample. The instrument blank chromatogram and quantitation report must be checked to insure it is within QC limits in Appendix C. It is also important to monitor the chromatographic baseline to insure there are no humps or disruptions which could be integrated as peak area when sample constituents elute on top of them. Surrogate recovery is not evaluated for IB samples. If the instrument blank meets these requirements sample analysis may proceed.

9.3 Batch QC

9.3.1 Method Blank

- A method blank (MB) is extracted and analyzed with each extraction batch to demonstrate that the entire analytical system - from extraction through GC analysis - is free of contamination.

- Analyze the MB according to Section 8.

- Evaluate the MB as soon as possible after it has been analyzed to determine if the results are within QC limits. See Appendix C for QC limits.

- Corrective action - If the MB is not acceptable, the source of the contamination must be found and eliminated and the problem documented before analysis can proceed. If re-analysis does not solve the problem, the batch may have to be re-extracted. Corrective action is decided by the EPA TOPO on a case by case basis.

- If the surrogate recovery does not meet acceptance criteria, re-analyze the extract. If the surrogate recovery fails high and the analytes in the blank are $\leq$ ½ the QL, report and narrate. If the surrogate fails low, all samples in the batch that are not ND may have to be re-extracted. Corrective action is decided by the EPA TOPO on a case by case basis.

9.3.2 Laboratory Control Sample

- Analyze a laboratory control sample (LCS) to demonstrate that the analytical system is in control. A LCS is extracted and analyzed once per extraction batch. The LCS is a MB spiked with laboratory fortified matrix solution.

- Analyze a LCS containing the target fuel at a concentration of 2,500 µg/L for water or 50 mg/kg for soil according to Section 8 of this SOP.

- Calculate the percent recovery (%R) using Equation 7.

- The %R must be within the QC limits in Appendix C. If acceptable accuracy cannot be achieved, the problem must be located and corrected prior to reporting any sample data and before additional samples are analyzed.

9.3.3 Matrix Spike/Matrix Spike Duplicate

- Laboratory fortified matrix (MS) and duplicate (MSD) samples are extracted and analyzed for each extraction batch. Matrix QC samples are usually designated in the field. In the event that a sample was not designated as the laboratory fortified matrix spike sample and adequate sample volume exists, the analyst will choose one representative sample from the SDG for QC analysis. The analyst shall not designate any obvious field blanks as the QC sample.

- Analyze the MS/MSD extracts according to Section 8 of this SOP as soon as possible following the analysis of the sample designated as the laboratory fortified matrix sample.

- Calculate the recovery of each compound using Equation 7.

  Equation 7:

  $$\% \text{ Rec} = ((SSR - SR)/SA) \times 100$$

  Where,
  SSR = Spiked sample result
  SR = Sample result
  SA = Spike added

- Calculate the relative percent differences (RPD) of the recoveries of each compound in the MS and MSD using Equation 8.

  Equation 8:

  $$RPD = \left| \frac{(MSC - MSDC)}{(MSC + MSDC)/2} \right| \times 100$$

Where,
MSC = Measured concentration of analyte in MS
MSDC = Measured concentration of analyte in MSD

- See Appendix C for QC limits.

  The MS/MSD recovery limits are advisory limits only. If the limits are not met, no further action is required, as long as the LCS is within limits, since the purpose of these analyses is to determine matrix effects on compound recovery. However, frequent failure to meet the recovery or RPD criteria should alert the analyst that a problem may exist and must be investigated. The analyst should analyze the matrix spike solution and check the recoveries of the spike compounds. A new solution should be prepared if the recoveries are not within 20% of expected.

- The table below lists the action to be taken based on the LCS and MS/MSD results.

QC ACCEPTANCE MATRIX + = PASS ! = FAIL								
CASE	1	2	3	4	5	6	7	8
BS - % REC	+	+	+	+	!	!	!	!
MS/MSD -% REC	+	!	+	!	+	!	+	!
MS/MSD - RPD	+	+	!	!	+	+	!	!

Case 1: Extraction batch acceptable.

Case 2: Extraction batch acceptable; matrix effect confirmed.

Cases 3 & 4: Extraction batch is unsatisfactory. Investigate MS/MSD problem and document findings in report narrative.

Case 5: Extraction batch rejected. Batch may have to be re-extracted unless LCS problem is determined and documented.

Cases 6, 7 & 8: Extraction batch rejected. Re-extract batch.

9.4 Sample QC

9.4.1 Surrogate Recovery

- Calculate the surrogate recovery in all field and QC samples immediately after analysis using the following formula:

Equation 9:

$$\%R = (\text{Amount Found}/\text{Amount Spiked}) \times 100.$$

- The surrogate recovery must be within QC limits. See Appendix C for QC limits.

- Take the following steps if surrogate recovery is not within the limits:

  1. Ensure that there are no calculation errors, and check the system performance.
  2. Re-analyze the extract if a system performance problem or calculation error is not evident. The extract may be diluted for re-analysis if examination of the chromatogram so indicates.
  3. If re-analysis of the extract does not solve the problem, the sample may have to be re-extracted. Corrective action is decided by the EPA TOPO on a case by case basis.

- Do not re-extract undiluted samples with surrogate recoveries outside the limits if the diluted analysis with acceptable surrogate recoveries is being submitted. Report the event in the run log.

- Do not re-analyze the MS or MSD samples, even if surrogate recoveries are outside the limits.

- If the sample associated with the MS/MSD analyses does not meet the surrogate recovery criteria, it should be re-analyzed only if the matrix spike and duplicate surrogate recoveries are within the limits. If the sample and spikes show the same pattern (i.e., outside the limits), then the sample does not need re-analysis. The similarity in surrogates recoveries in the sample and spike analyses must be discussed in the report narrative.

- If the surrogate recoveries of the re-analysis of the extract are within limits, then:

  1. If the re-analysis was undiluted, the problem was within the laboratory's control. Report the results from the re-analysis and submit the data from both analyses. Distinguish between the analysis and re-analysis by

adding an "RE" suffix to the client sample ID on the re-analysis. The problem must be documented in the report narrative.

2. If the re-analysis was diluted, the problem was a matrix effect. Report the results from the re-analysis and submit the data from both analyses and discuss the result in the report narrative. The problem must be documented in the report narrative.
3. If the surrogate recoveries of the re-extraction are within limits, then the problem was within the laboratory's control. Report the results from the re-extraction. Distinguish between the original analysis and the re-analysis by adding the "RE" suffix to the client sample ID in the re-analysis. The problem must be documented in the report narrative.

- If the re-extraction does not solve the problem, report the results from the first analysis and submit the data from both analyses. Distinguish between the original analysis and the re-analysis by adding the "RE" suffix to the client sample ID in the re-analysis. The problem must be documented in the report narrative.

9.5 Method Performance

Region 9 Laboratory performance for this procedure from is summarized in the following table.

Method Performance

Analyte	Matrix	QC Type	Number of Measurements	Mean Recovery, %	95% Confidence Interval (2σ)
Diesel	Water	LCS	55	84.6	64 – 105
Diesel	Solid	LCS	75	81.9	66 - 97

Note: water data selected from June 2009 to May 2010; solid data selected from September 2009 to April 2010.

The following functional areas of the SOP may be significant sources of analytical error:

- Poor extraction efficiency due to specific analyte characteristics or other problems.
- Standard degradation
- Chromatographic separation and peak integration.

10 DOCUMENTATION

10.1 Standards

All standards (ICAL, ICV/CCV, QL, MS/MSD, and LCS) are recorded in the Element database. A copy of each Analytical Standard Record associated with sample analysis must be included in the data package.

10.2 Analytical sequence

Document the analytical sequence in the Element database.

Record the instrument ID and the LIMS calibration ID for each sequence. Record the Lab number, analysis, position, and LIMS standard ID, as applicable for each field and QC sample in the Element analysis sequence.

10.3 Analytical Report and Data Package

Analytical reports are produced using the Element database. The data package is produced from Element database and manual log records. Appendix G provides the typical format for data package deliverables.

10.4 Maintenance Logbook

Maintain a maintenance logbook for each instrument. Whenever corrective action is taken, record the date, the problem and resolution, and documentation of return to control. Document all preventive or routine maintenance performed, as well as repairs or corrective or remedial actions in accordance with EPA Region 9 Laboratory SOP 840, *Notebook Documentation and Control*.

10.5 SOP Distribution and Acknowledgement

Distribute the approved SOP to all laboratory staff expected to perform the SOP or review data generated by the SOP. The Lab QC Database is used to maintain the list of assigned analysts for each SOP. Analyst training is documented via the Training Record form and the Read and Understood Signature log; the latter is entered into the Lab QC Database.

10.6 SOP Revisions

Revisions to this SOP are summarized in Appendix H.

11 REFERENCES

Agilent Technologies 6890 Gas Chromatograph Users Manual

Agilent Technologies EnviroQuant ChemStation User's Guide

U.S. Environmental Protection Agency, *Method 8000C, Determinative Chromatographic Separations, Revision 3, March, 2003.*

U.S. Environmental Protection Agency, *Method 8015C, Nonhalogenated Organics Using GC/FID, Revision 3, Feb. 2007.*

U.S. EPA Region 9 Laboratory SOP 125, *Disposal Procedures for Unused Aqueous Environmental Samples.*

U.S. EPA Region 9 Laboratory SOP 275, *Extraction of Water Samples by Continuous Liquid-Liquid Extraction.*

U.S. EPA Region 9 Laboratory SOP 290, *Extraction of Soil Samples Using Pressurized Fluid Extraction.*

U.S. EPA Region 9 Laboratory SOP 706, *Laboratory Waste Management Procedures.*

U.S. EPA Region 9 Laboratory SOP 805, *Refrigerator Temperature Monitoring*

U.S. EPA Region 9 Laboratory SOP 835, *Chromatographic Integration Procedures*

U.S. EPA Region 9 Laboratory SOP 840, *Notebook Documentation and Control*

U.S. EPA Region 9 Laboratory SOP 880, *Demonstration of Laboratory Capability and Analyst Proficiency*

APPENDIX A.
DEVIATIONS FROM THE REFERENCE METHOD

A.1 Deviations

1. In the SOP, the retention time range for Diesel Range Organics is C10 to C24, not the retention time of C_{10} and C_{28} alkanes as specified in the reference method. In addition, the SOP extends the chromatographic range of the method to include Oil Range Organics C24 to C40, as an analyte.

2. The CF is area/concentration unit (µg/mL) not area/mass (ng) as in the reference method. The formulas for determining sample analyte concentrations have been modified to reflect this change.

APPENDIX B.
ANALYTES AND QUANTITATION LIMITS

Hydrocarbon Fuel	QL, on column, µg/mL	QL, Solid, mg/kg (30g sample)	QL, Water, µg/L (1 L sample)
DRO: C10-C24	50	5	250
ORO: C24-C40	200	20	1,000

APPENDIX C.
CONTROL MEASURES AND CRITERIA

QC MEASURE	CRITERIA
Initial Calibration (ICAL)	RSD < 20
Second Source Verification (SCV)	Analyze after ICAL. CF within 30% of mean ICAL CF
Calibration Verification (CCV)	Analyze before QC or field samples and every 12hrs, or more frequently, thereafter. Results: %D ±20
Quantitation Limit Standard (QLS)	Analyze each day that field or QC samples are analyzed. Result: ± 40% of true value
Method Blank (MB)	Extracted once per extraction batch or every 20 samples, whichever is more frequent. Results must be < ½ QL of target analytes.
Instrument Blank (IB)	< ½ QL of target analytes
Laboratory Control Sample (LCS) fortified with Diesel	Extracted once per extraction batch. Result: %R between 54 and 115 for water and 59 and 110 for solids.
MS/MSD fortified with Diesel	Extracted once per extraction batch. Result: %R between 67 and 110 for water and 20 and 153 for solids; RPD less than 20% for water and 50% for solids.
Surrogate Recovery of QC and field samples (except IB)	%R between 60 and 127 for water and 41 and 148 for solids.

Note: water limits are based on data from June 2009 to May 2010 to use a significant quantity of data; solid limits for LCS and MS/MSD are from September 2009 to April 2010 to reflect changes in the preparation SOP 290 Revision 5. Surrogate data for solids selected from September 2009 to January 2010 to exclude non-typical samples analyzed during February to April 2010 which were not representative of method performance.

APPENDIX D.
RECOMMENDED INSTRUMENT OPERATING PARAMETERS

Instrument: Agilent 6890
Chromatographic column: 15m x 0.32mm ID, 0.1µm film (Restek Rtx-1)

OVEN	
Maximum temperature:	350°C
Equilibration time:	0.50 min.
Initial temperature:	50°C
Initial time:	2.00 min.
Ramp:	
Rate 1:	15.00°C/min
Final temperature 1:	325°C
Final time 1:	14.00 min.

INLET	
Mode:	Pulsed splitless
Temperature:	320°C
Pressure:	3.00 psig
Pulse pressure:	10.0 psig
Pulse time:	0.30 min
Purge flow:	60 mL/min.
Purge time:	0.30 min.
Gas saver:	On
Gas saver flow:	20.0 mL/min
Gas saver time:	2.00 min.
Carrier gas:	Helium

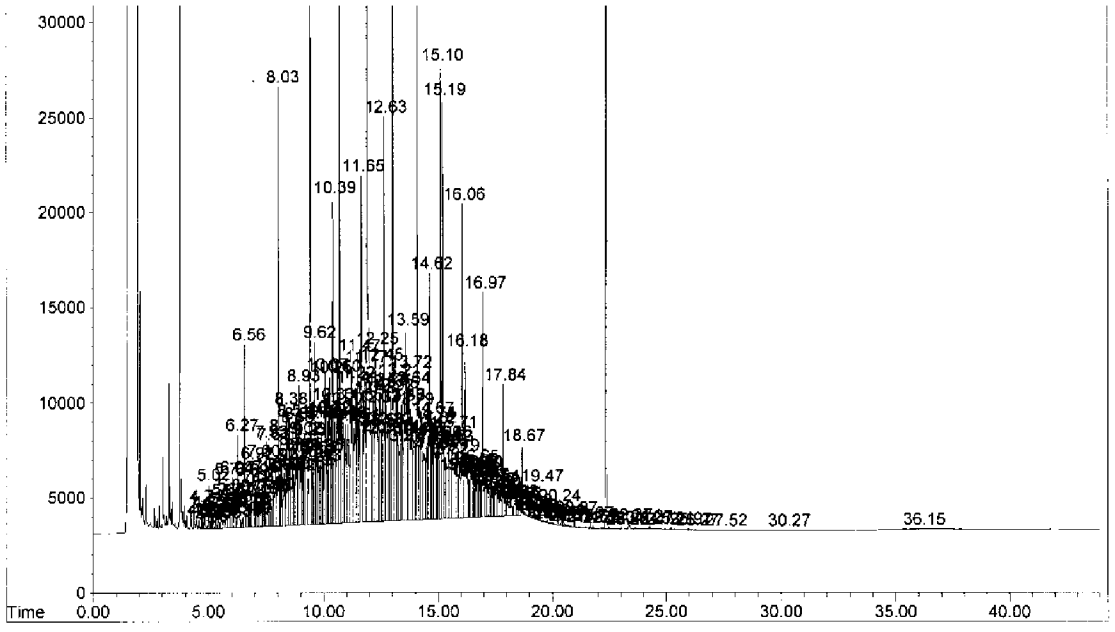
COLUMN	
Mode:	Ramped pressure
Initial pressure:	3.00 psig
Initial time:	2.00 min.
Rate 1:	0.61 psig/min.
Final pressure 1:	20.00 psig
Final time 1:	0.13 min.
Rate 2:	20.00 psig/min.
Final pressure 2:	30.00 psig
Final time 2:	5.00 min.
Nominal initial flow:	1.1 mL/min.
Average velocity:	21 cm/sec

DETECTOR (FID)	
Temperature:	350°C
Hydrogen flow:	40 mL/min.
Air flow:	440 mL/min.
Mode:	Constant makeup flow
Makeup flow:	49.0 mL/min
Makeup gas:	Nitrogen

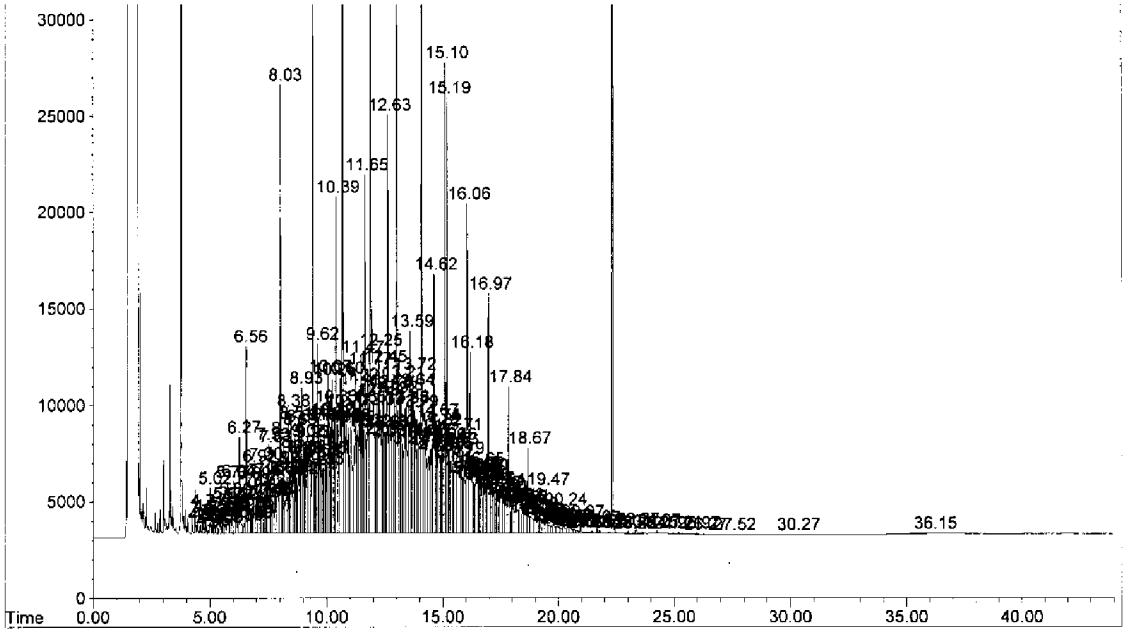
SIGNAL	
Signal:	Signal - Col Comp
Data rate:	50 Hz
Start save time:	1.80 min.
Stop save time:	30.00 min.
Column Comp:	Off

INJECTOR (7673)	
Sample washes:	1
Sample pumps:	3
Injection volume:	2.0 microliters
Syringe size:	10 microliters
PostInj Solvent A washes:	3
PostInj Solvent B washes:	3
Viscosity delay:	0 seconds
Plunger speed:	Fast
Pre Injection dwell:	0.00 min.
Post Injection dwell:	0.00 min.

APPENDIX E.
INTEGRATION EXAMPLES



INCORRECT BASELINE INTEGRATION



CORRECT BASELINE INTEGRATION

APPENDIX F.
PREVENTIVE MAINTENANCE REQUIREMENTS

Item	Frequency	Actions/Comments
Split vent trap	As Needed	Replace.
Flowmeter calibration	2 years	Manual flowmeters only.
Syringes and/or syringe needles	As Needed	Replace syringe if dirt is noticeable in the syringe, if it cannot be cleaned, if the plunger doesn't slide easily, or if clogged. Replace needle if septa wear is abnormal or the needle becomes clogged.
Inlet liner	With each ICAL	Check often. Replace when dirt is visible in the liner or if chromatography is degraded.
Liner O-rings	With each ICAL	Replace with liner or with signs of wear.
Inlet septum	Daily (when analyzing samples)	Check often. Replace when signs of deterioration are visible (gaping holes, fragments in inlet liner, poor chromatography, low column pressure, etc.).
Inlet Hardware	As Needed	Check for leaks and clean. Check parts and replace when parts are worn, scratched, or broken.
Column Maintenance	With each ICAL	Remove 1/2-1 meter from the front of the column when experiencing chromatographic problems (peak tailing, decreased sensitivity, retention time changes, etc.).
Solvent rinse	As needed	When chromatography degradation is due to column contamination. Only for bonded and cross-linked phases.
Replacement	As needed	When trimming and/or solvent rinsing no longer return chromatographic performance.
Ferrules		Replace ferrules when changing columns and inlet/detector parts.
FID Jets & Collector	As needed	Clean when deposits are present. Replace when they become scratched, bent, or damaged, or when having difficulty lighting FID or keeping flame lit.
Purge/Sample Lines	As needed	Bake out and purge. Clean with organic free water if necessary.
Trap	As needed	Replace when loss of performance.

APPENDIX G.
TYPICAL DATA PACKAGE FORMAT

Data package contents, in order. Optional sections are shown in *italic text*. Separator pages are underlined.

Draft Report (from LIMS)

Data Package Cover [First numbered page in the data package]

Review Forms
- EPA Review Form
- ESAT technical review guide
- Discrepancy Reports (if applicable)
- Work Order Memo (if applicable)
- Daily folder review forms or checklists
- Analysis matrix listing all analytical runs (for organics only)

Tracking Forms
- Work Order(s)
- COC(s)

Sample Preparation (for projects that require extraction or digestion)
- Bench Sheets (and extraction logs, where used)
- Sample cleanup data and records (e.g. GPC logs)
- Moisture data as applicable

[Analysis Method] Data (For each method where multiple methods in package)
- Bench sheet(s) where not used in Sample Preparation section
- Sequence logs and instrument or other data as applicable, in run order and grouped by day.

Alternatively, separate calibration and sample data as:
- Initial Calibration Data
- Sample Data

Miscellaneous Data
- Other data as applicable (e.g. conductivity for perchlorate)

Standard Records
- Standards records from LIMS (and logbook pages as needed)

APPENDIX H.
REVISION HISTORY

STANDARD OPERATING PROCEDURE: 385
Revision: 4, Effective: 08/15/10

EXTRACTABLE PETROLEUM HYDROCARBONS BY GC/FID

Revision	Effective Date	Description
4	08/15/10	Changed reference method to EPA 8015C. Revised quantitation and reporting procedures.